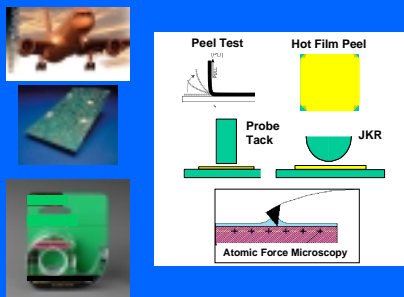




Motivation



“Bolts and screws can be modeled with software... , but glue makers have yet to come up with a predictive model”, Forbes, 10.29.01

The above quote explicitly states the challenge that is presented to our community. Polymer interfaces, whether they're created for photolithography, glues, or bacterial protection, define the efficiency and mechanical integrity of many of the technological applications being developed today. Unfortunately, our knowledge of interface formation and failure is relatively immature. We do know that interfacial strength is dependent upon numerous parameters including: surface energy, molecular weight, roughness, temperature, geometry, and humidity. Conventional investigations of polymer adhesion either produce non-quantitative results or the exploration of parameter space is inefficient. These roadblocks serve as the motivation for this project.

Objective

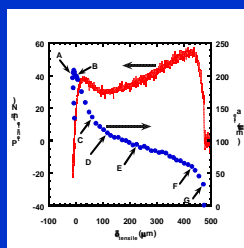
Develop methodology for quantitative high-throughput measurements of adhesive strength of polymer interfaces

Approach

Theory of Johnson, Kendall, and Roberts (JKR)

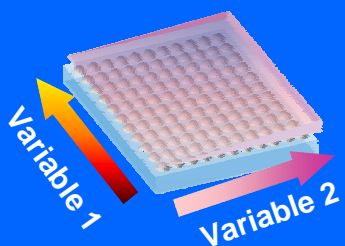


$$a^3 = \frac{9R}{16E} \left[P + 3\pi GR + \sqrt{6GRP + (3\pi GR)^2} \right]$$



The theory of Johnson, Kendall, and Roberts (JKR) was introduced in 1971. In its classical form, the theory states that by monitoring the applied force (P) and resulting contact area ($A = \pi a^2$) as a spherical punch contacts and separates a flat substrate, the interfacial energy (G) can be measured. This theory was constrained by two assumptions: 1) a was required to be much smaller than any other dimension in the contacting system, and 2) the materials must be linearly elastic. Although these assumptions restricted early use of JKR to weakly adhesive surface interactions, recent modifications to the theory have allowed a vast range of adhesive problems to be investigated, including the tack of strong adhesives such as PSA's (data on the left).

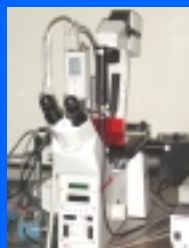
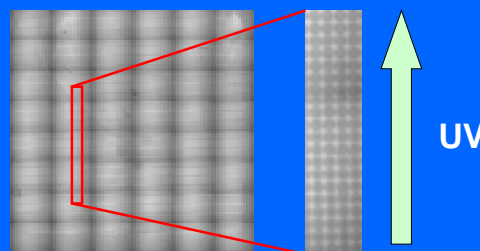
Combinatorial Design



- Measure a , δ
- Determine G

Possible Variables:

- Temperature
- Thickness
- Strain
- Surface Energy

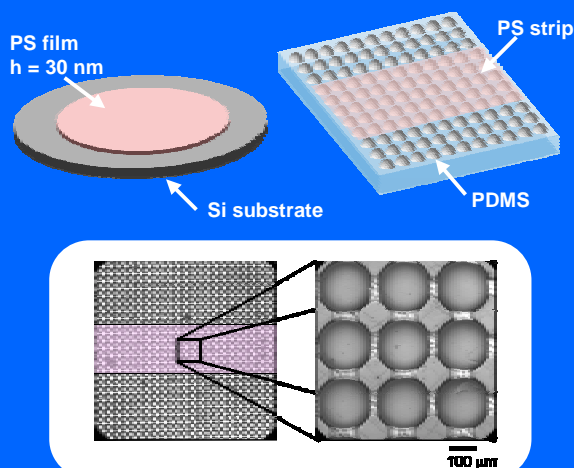


Although the JKR method yields a quantitative understanding of polymer interface formation and failure, the exploration of parameter space can be time consuming. To meet our objective, we proposed the development of a combinatorial JKR-type test involving an array of spherical lenses that is brought into contact with a complementary flat substrate. Established techniques of creating combinatorial gradient libraries can be used to modify either the complementary substrate or the array of lenses. By using gradient techniques, each contact point yields quantitative interfacial data for a unique combination of the two variables. Using the instrument on the left, this technique can potentially produce the same amount of data for thousands of conventional adhesion tests within minutes. The above data image shows the changing contact areas for a microlens array with approx. 8000 lenses in one square centimeter.

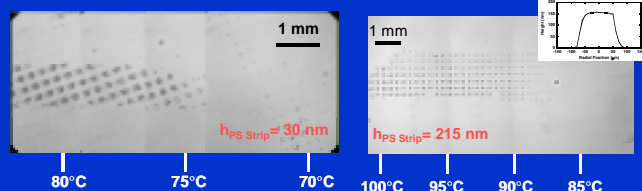
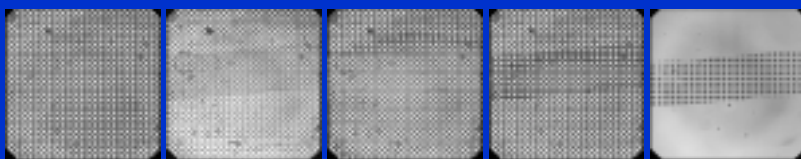


Library Design

To demonstrate our combinatorial methodology, we investigated the adhesion of thin glassy films and crosslinked elastomers. Specifically, we used a crosslinked PDMS (poly dimethylsiloxane) microlens array that was fabricated through a molding technique in the same manner that stamps for soft lithography are made. Onto this PDMS microlens array, we floated a thin film coating of polystyrene. Our flat substrate was a silicon wafer that was coated with a uniform film of atactic polystyrene (MW = 114, 000 g/mol). For initial tests, we only coated a central region of our PDMS array. This created two distinct interfacial regions in our library: PS/PS interfaces in the center of our array and PS/PDMS interfaces on either side.



Library Evaluation

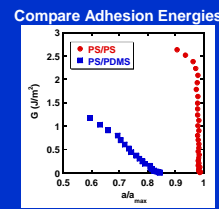
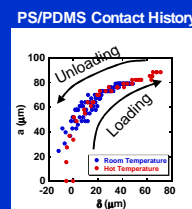
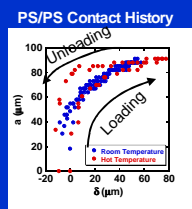
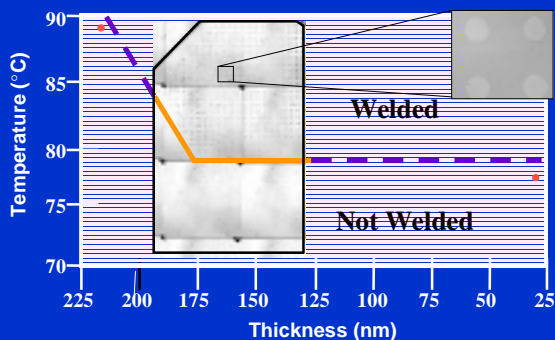


Time-Temperature dependence for interfacial strength development indicated by changing weld spot size and thickness dependence.

The sequence of images at the top (left) shows the microlens array contacting and retracting from the PS-coated Si wafer at a temperature of 80°C. This sequence of images demonstrates the qualitative mapping of adhesion that can be accomplished simply by watching the contact history of the microlens arrays. The microlenses in the strongest region of contact separate last.

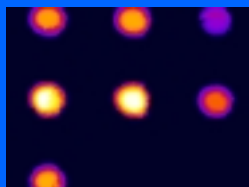
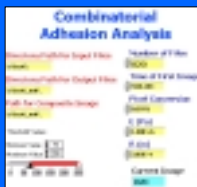
At elevated temperatures, the increased adhesion of the PS/PS interfaces leads to coating failure and causes "weld" spots to be deposited on the PS-coated Si wafer. We used a temperature gradient, and ultimately a library of temperature and film thickness, to map the critical temperature for coating failure.

Analyzing the contact history of each microlens yields quantitative data of the interfacial failure process as indicated by the data below. We find all "weld" spots fail at an applied energy release rate of 4.7 J/m².



Same Sample, Same Conditions!

Informatics



To complete the combinatorial methodology, we have developed software to demonstrate how the contact histories of each microlens can be analyzed in an automated manner to produce adhesion maps. The software is written using National Instrument's Labview programming language with the ultimate goal of marrying instrument control with on-line analysis. The map on the left uses color to represent time of contact for a series of PDMS microlenses at fixed displacement.



Surface Forces

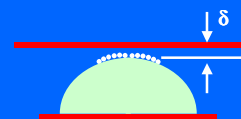
As two surfaces approach each other, a critical distance exists at which the surface forces will overcome the elastic restoring forces of the material system. At this distance, the two materials will spontaneously create an interface. Quantifying these surface force interactions is important for biological systems, colloidal dispersions, and MEMS devices. In this poster, we present our work using an alternative form of the microlens contact adhesion test to quantify the effect of surface energy on the dynamics of interface formation.

JKR "Zero Force" contact radius

$$a_0 = \left(\frac{27\pi R^2 G}{8E} \right)^{1/3}$$

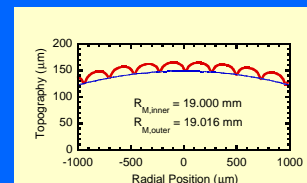
Lens Arrays

Crosslinked Polydimethylsiloxane (PDMS)
 $E = 1.0 \text{ Mpa}$; $\gamma \sim 20 \text{ mJ/m}^2$



Substrates

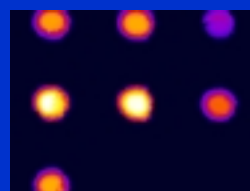
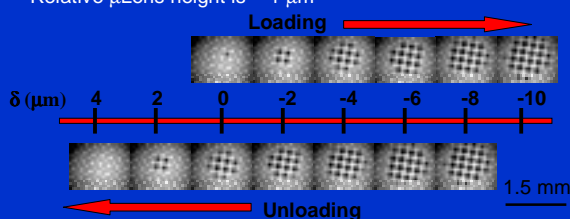
Bare glass slide
 $\gamma = 67 \text{ mJ/m}^2$
n-octyl dimethylchlorosilane coated glass slide
 $\gamma = 28 \text{ mJ/m}^2$
Fluorinated glass slide
 $\gamma = 10 \text{ mJ/m}^2$



Controlling Separation

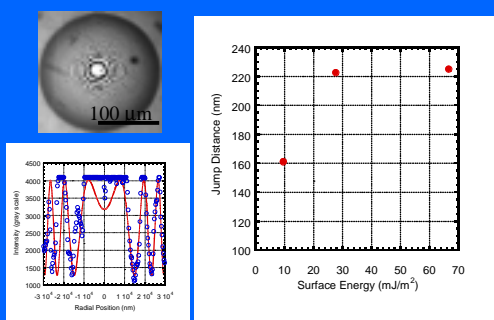
$R_{\text{macro}} = 19.0 \text{ mm}$
Relative μLens height is $\sim 1 \mu\text{m}$

$R_{\text{macro}} = 200 \text{ mm}$
Relative μlens height is 100 mm

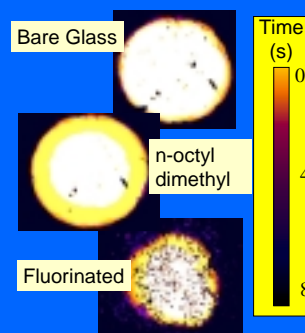


To initially bracket the range of surface interactions, we created a macrolens integrated with an array of microlenses on the surface. The curvature of the macrolens naturally creates a gradient in separation distances as the macrolens approaches a substrate. This data (on left) shows contact histories for two macrolenses of different curvatures. For the smaller curvature lens, a single contact of a microlens is stable since the neighboring microlenses are approximately $1 \mu\text{m}$ away from the surface. For the larger curvature lens, the neighboring microlenses creep into contact over time at fixed displacement since they are within the critical separation distance.

Quantifying Attraction



In addition to using the curvature of a macrolens to measure the critical separation distance, we use our precision motion control system and high resolution optics to accurately measure the exact separation distance by analyzing the interference rings under a single microlens. As indicated by the graph on the right, the substrate surface energy weakly affects the critical separation distance. Although this data can be collected with an AFM, we have an advantage of monitoring the dynamics of the contact process visually. The contact history images (color represents time) on the right demonstrate that the local surface structure changes the rate of interface formation.



Substrate	Pull-in Force (μN)	Pull-in Stress (MPa)
Bare glass	20	5.8×10^{-3}
n-octyl dimethyl	20	5.8×10^{-3}
Fluorinated	9	6.3×10^{-3}